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Method of manufacturing ceramic tooth restorations

5 The present invention relates to powder metallurgical manufacturing methods of accurate-to-shape tooth restorations with an individually manufactured core in a densely sintered, high strength ceramic material, which fits against prepared tooth surfaces or artificial abutments. On these cores dental porcelain can be fired to tooth crowns, inlays, veneers or bridges.

10 Swedish patent application 8900620-9 discloses a method of manufacturing ceramic bodies with irregular shapes where the sintering shrinkage is considered.

US 5,080,589 discloses a method of manufacturing copings in densely sintered high strength ceramic material where the sintering shrinkage is considered. According to this patent the copings are premanufactured, which means that they have in advance given dimensions.

According to US 5,106,303 and Swedish patent application 9002959-6 inlays, onlay crowns and veneers can be manufactured in densely sintered high strength ceramic material by copy milling a green body or a presintered or sintered body from an impression of the prepared tooth surface considering the sintering shrinkage.

20 The object of the present invention is to provide rational manufacturing technique for manufacturing tooth crowns, inlays, veneers or bridges in densely sintered, high strength ceramic material by using modern powder metallurgical technique, reading technique and machining technique.

25 The present invention relates to a method of manufacturing artificial tooth restorations for natural teeth or implants containing an individual core in densely sintered, high strength ceramic material with dental porcelain fired to its outer surface. The inner surface of the core, which will fit to one or more prepared surfaces or

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artificial abutments is manufactured by forming a ceramic powder mixture against a surface of a body which surface is manufactured by registering with a three dimensional optical or mechanical reading method the prepared surfaces of the prepared teeth or artificial abutments and their mutual relationship, either directly in the mouth or on a model in e.g. plaster after which the registered surfaces are reproduced in an enlarged size into a carrier e.g. by using a computer controlled milling machine, the enlargement being calculated considering the shrinkage of the ceramic material during the sintering to full density with the addition of a desired gap for cement. The carrier is made from a model material e.g. plaster, graphite or a polymer.

In some cases the outer surface of the core can be surrounded by prepared tooth surfaces e.g. inlays. The core is manufactured in this case by first compacting a ceramic powder against a carrier, which partly has an outer shape, which is smaller than the outer surface of the core, which will fit against the prepared tooth surfaces. Then the outer shape of the core is made by milling the green body in the milling machine. In order to make it possible to machine the outer surface, the prepared surfaces are registered and their mutual relationship determined with a three dimensional optical or mechanical scanning technique, either directly in the mouth or on a model in some model material e.g. plaster or on an impression with some impression material e.g. addition-silicon or a wax impression, from the prepared surfaces. The registered surfaces are reproduced in an enlarged size onto a carrier, e.g. with a computer controlled milling machine, which machines the powder compacted against the carrier. The enlargement is calculated from the sintering shrinkage of the ceramic material upon sintering to full density. In this case a reduction is made for desired gap for cement.

Fig. 1 shows cross sections of natural tooth one with an artificial tooth crown (Fig. 1a), one with an inlay (Fig. 1b), and one with a veneer (Fig. 1c). In this figure A= dental porcelain, B= core, Y= outer surface of the core, I= inner surface of the core which will fit against the prepared surface, C= cement, P= prepared surface of the tooth, S= preparation border, E= enamel, D= dentine and F= pulp.

Fig. 2 shows a cross section of a bridge, which is cemented on two supporting teeth. These supporting teeth can have a vital abutment (U<sub>1</sub>) or an artificial abutment (U<sub>2</sub>) manufactured in a dental alloy, ceramic material or a strengthened polymer. The bridge in this figure contains two artificial dental crowns according to Fig. 1a and with a pontic (V) between as replacement for a lost tooth. The bridge contains a core (B) with dental porcelain (A). A bridge can contain more supporting teeth and also more pontics in between. The supporting teeth can also be prepared for inlays or veneers. Veneers can be made both on buccal and lingual surfaces. The supporting teeth can also be implants with artificial abutments.

Figs. 3a and b show a cross section of a cylindrical blank K<sub>1</sub> manufactured in e.g. plaster, graphite or a polymer. K<sub>1</sub> has the diameters  $\Phi_1$  and  $\Phi_2$ , and a groove Q<sub>1</sub> for repositioning in a chucking fixture in a milling machine and a bevel Z<sub>1</sub>.

Fig 3c shows K<sub>0c</sub>, a model of a tooth prepared for a crown. P is the prepared tooth surface and S the preparation border.

Fig 3d shows K<sub>2</sub>, is a cross section of a reproduction with enlarged size of K<sub>0c</sub> produced in a computer controlled milling machine from a blank K<sub>1</sub>. The enlargement is calculated considering the sintering shrinkage of the ceramic material. L<sub>2</sub> is a reproduction of the prepared

tooth surface (P) and is defined as the surface above the preparation border S<sub>2</sub>.

Fig 3e shows K<sub>3</sub> which is K<sub>2</sub> with a shell of 1-1,5 mm outside the enlarged surface (L<sub>2</sub>). L<sub>3</sub> is the outer surface of this shell. L<sub>3</sub> is finished with the border S<sub>3</sub>.

Fig. 4 shows a cross section of an example of a press for individual cores for tooth restorations. F is a cylindrical tube. G is a cylindrical plate with the diameters  $\Phi_3$  and  $\Phi_4$ . H is a cylinder with diameter  $\Phi_3$ . H, F, and G can be made in e.g. hardened steel E is made in a viscous elastic material as well as J and X. X is an impression from K<sub>3</sub> except the part with the diameter  $\Phi_1$ . X has a cylindrical outer shape with diameter  $\Phi_3$ . In Fig. 4 K<sub>2</sub> has been placed in X, M is a cavity with space for ceramic powder and P<sub>K</sub> is the force with which H is pressed against G.

Fig. 5a shows K<sub>0i</sub>, which is a cross section of an impression of an inlay. The surface P<sub>i</sub> is an impression of the prepared surface of the inlay (P) (Fig. 1b) and S is the preparation border. Figs. 5b and 5c show a cross section of K<sub>4</sub> (Fig 5b) and K<sub>5</sub>(Fig 5 c), which are used for manufacturing of cores for inlays. K<sub>4</sub> is a model of K<sub>0i</sub> enlarged considering the sintering shrinkage. K<sub>4</sub> can be made by milling in a computer controlled milling machine from a blank K<sub>1</sub> in plaster, graphite or a polymer. In this figure the surface L'<sub>4</sub> (broken line) has been drawn. L'<sub>4</sub> is the enlarged surface of the inlay, which exactly fits against the prepared surface (P) of the tooth prepared for an inlay. S<sub>4</sub> marks the preparation border. L<sub>4</sub> is the milled surface of K<sub>4</sub>. The distance between L<sub>4</sub> and L'<sub>4</sub> is equal to the thickness of the core in unsintered condition. K<sub>5</sub> consists of K<sub>4</sub> with a shell of 1,5 - 3 mm outside the continuous surface L<sub>4</sub>. L<sub>5</sub> is the outer surface of this shell.

Fig. 6 shows  $K_{ob}$ , which is a cross section of a model of two supporting teeth  $c_1$  and  $c_2$  prepared for crowns and intermediate gingiva.

Fig. 7 shows  $K_6$ , which is an enlarged model of  $K_{ob}$ .  
5 The enlarged supporting tooth of  $K_6$  are  $C_1$  and  $C_2$ . The model can be cut at the lines  $l_1$  and  $l_2$ .

Fig. 8 shows the enlarged model from Fig. 7 with a shell with outer contour  $L_8$ , which surrounds the preparations  $C_1$  and  $C_2$  and intermediate gingiva.

10 As can be seen from Fig. 1 and Fig. 2 artificial tooth crowns, inlays, veneers or bridges are made as a core in densely sintered ceramic (B) with dental porcelain (A) fired to the outer surface (Y) or the core. The tooth restorations are fixed to the prepared surfaces (P) by e.g. adhesive (cement), as well as the bridge to the abutments  $U_1$  and  $U_2$ . The thin layer of cement (C) connects the prepared  
15 tooth surface (P) with the inner surface of the core (I), which fits with desired precision to the prepared surface (P). On the outer surface (Y) of the core (B) dental porcelain can be fired. The layer of cement can have a thickness  
20  $< 100 \mu m$ , preferably  $25-50 \mu m$ . The cement thickness can at some spots be  $> 100 \mu m$  depending on small irregularities in the preparation. The cementing of the restorations can be made with e.g. phosphate cement, glass ionomer cement or  
25 some resin cement in accordance with known techniques. In the latter instance, it can be an advantage to silane treat the inner surfaces (I) of the cores of the constructions, which are to be joined with the prepared surfaces (P). It also can be advantageous to etch the prepared surface (P),  
30 which consists of enamel (E) before the cementation of the veneer with resin cement. The etchant can be phosphoric acid. Parts of the prepared surface consisting of dentine can be treated with some bonding resin before the cementation.

The ceramic powder can be made by several well known methods. Traditional powder metallurgical technique can be used, where the different components are mixed and ground under dry or wet conditions with water or an inorganic solvent (e.g. alcohol) as grinding liquid. To the ceramic  
5 slurry lubricants or other organic binders are added, when needed at suitable time in the process in accordance with known techniques.

The ceramic base material in the core is suitably one  
10 or several biocompatible oxides, alternatively with additives of carbides and nitrides with or without binders. Examples of biocompatible oxides, which can form the base matrix for the ceramic body, are  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{MgO}$ ,  $\text{ZrO}_2$  and  $\text{ZrO}_2$  with additives of smaller amounts of up to 10 mole% of  
15  $\text{Y}_2\text{O}_3$  or  $\text{MgO}$  (partly or totally stabilised  $\text{ZrO}_2$ ). Included components can be present as particles with a size of  $<25 \mu\text{m}$ , preferably  $<10 \mu\text{m}$ , and/or as whiskers (hair shaped single crystals) with a length of  $>10 \mu\text{m}$ , preferably  $>25 \mu\text{m}$ ,  
20 and a length to diameter ratio  $>5$ , preferably  $>10$  and/or fibres (polycrystalline) with a diameter  $>10 \mu\text{m}$  and/or as single crystal platelets with an approximate diameter of 5-  
50  $\mu\text{m}$  and a thickness of 1-10  $\mu\text{m}$ . The amount of whiskers, fibres and/or platelets should not exceed 60 volume%. In a preferred embodiment the ceramic material comprises  $>50\%$   
25  $\text{Al}_2\text{O}_3$  with additives of conventional sintering aids. In order to increase the strength  $<25$  weight %, preferably 3-12 weight %, of  $\text{ZrO}_2$ , and/or 5-40 weight%, preferably 10-30 weight%, of  $\text{SiC}$ -,  $\text{TiN}$ - or  $\text{ZrN}$ -whiskers can be added. It is  
30 important that the ceramic material is sintered to closed porosity, which for an oxide material means at least 98% of theoretical density, but in order to ensure good mechanical strength the material should preferably have a density over 99%, while densities over 99.5% give the best strength.

In order to get a suitable colour of the core (B) coloured components can be chosen. Additives e.g. 0.1-1 weight  
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% of TiN and/or ZrN will give  $\text{Al}_2\text{O}_3$  based cores a faint yellow shade.  $\text{Fe}_2\text{O}_3$  gives a yellow brown shade and  $\text{MnO}_2$  gives a rose-coloured shade. Of course additives, which give unesthetic effects, should not be used.

5       According to the present invention artificial ceramic tooth crowns, inlays, veneers or bridges can now be made from a ceramic powder with additions of lubricants and/or other organic binders. When manufacturing the core of an individual tooth crown, the ceramic powder is compacted  
10       against the body  $K_2$  to a core, which after sintering to full density fits to the prepared tooth surface and with an as close to desired wall thickness as possible. When manufacturing the enlarged model ( $K_2$ ) the sintering shrinkage has to be considered by enlarging the surface ( $L_2$ ) so that  
15       the compacted body during the subsequent sintering process to full density, with great accuracy will have desired final, inner geometrical shape (I), which fits against the prepared surface (P). The desired shape of the gap between the tooth crown and the prepared tooth for cementing the  
20       crown to the prepared tooth must also be considered. The size of this gap can be calculated e.g. to be as small as possible at the preparation border preferably as close to zero as may be achieved during the manufacturing process.

      The manufacturing of an individual core of a tooth  
25       crown can be made in the following way: A body  $K_2$  is manufactured from a blank  $K_1$  by registering a prepared tooth surface with a three dimensional optical or mechanical reading method. The registered surface is transferred to a computer controlled milling machine, which gives the shape  
30       of  $K_2$  based on the data from the registration and data about the sintering shrinkage and desired shape of gap for cement. A body  $K_3$  is manufactured from the above registered surface, with the addition of a shell of 1,5 - 3 mm according to Fig. 3e. The tool X is manufactured by placing  $K_3$  in  
35       a mould in which a viscous elastic material e.g. an Addi-

tion-silicone, which is allowed to harden. The tool X can also be made using the blank  $K_1$  or a somewhat reduced  $K_1$  instead of  $K_3$ . When using  $K_1$  to manufacture X the amount of ceramic powder will increase and at the same time more compacted powder has to be milled. The advantage is that this tool X can be used for different sizes of preparations and the bevel  $Z_1$  is not needed. Ready to press ceramic powder is placed in the cavity (M) of X and the body  $K_2$  is placed in X. It can be an advantage to treat the surface  $L_2$  of  $K_2$  with a thin layer of an adhesive material to increase the adhesion of the ceramic powder to  $K_2$ .  $K_2$  is fixed with help of the bevel  $Z_1$ . J is placed on  $K_2$  and X with content in the tube F. E is put in place as well as the cylinder H whereupon the tube F with content is placed on the cylindrical end plate G. The whole tool according to Fig. 3 is placed in a uniaxial press e.g. a hydraulic press. During the compaction of the powder the cylinder H is pressed with the compaction force  $P_K$  into the tube F against the cylinder end plate G in contact with the elastic material J. During pressing, the powder is compacted against the body  $K_2$ . After the compaction, H, J and  $K_2$  are removed with compacted powder, which adheres to  $K_2$ . If a dry bag isostatic press is used the tool X is not needed at all. The ceramic powder is just compacted against the body  $K_2$  treated with an adhesive material.

After the compaction of the ceramic powder against  $K_2$  this carrier is repositioned in the computer controlled milling machine. The groove  $Q_1$  and to this matching pin in the chucking fixture of the milling machine makes it possible to reposition in direction of rotation and the step between the diameters  $\Phi_1$  and  $\Phi_2$  in axial direction. The outer surface of the coping is machined after repositioning in the milling machine. The outer surface can be calculated by the computer, which controls the milling machine. The outer surface can also be made from data of a scanned

coping, which is modelled in e.g. wax and which before the scanning is fixed on the model of the prepared tooth. The machining can be controlled by the computer so that the coping gets the desired outer form and be adapted to the preparation border. The shell thickness of the core can be 0,3 - 1 mm, however, in certain places shell thicknesses >1 mm can be needed for strengthening or other reasons. It is also possible from data of contact conditions with neighbouring teeth in the same or opposite jaw to machine a complete ready tooth or from scanned data from a crown, which is modelled in e.g. wax and which before the scanning is fixed on the model of the prepared tooth. During all machining in the milling machine the sintering shrinkage is taken into account.

The compacted body is presintered on the carrier K<sub>2</sub> manufactured in the milling machine. For alumina the presintering will be at 800°C - 1000°C on the carrier. During the presintering the model material (plaster) will dehydrate, which results in a shrinkage of about 15%. The ceramic material will shrink just a few percent during presintering. The presintered core will, thus, be too large on the plaster model and can be removed and be sintered to full density. Instead of plaster investment materials can be used provided that they have enough strength and will not deform during the powder compaction. The carrier K<sub>2</sub> can also contain a material e.g. graphite or a polymer which will burn slowly during the presintering period. It is important that the thermal expansion coefficient of the carrier material is less than the thermal expansion coefficient of the ceramic material, and the adhesive material must disappear during presintering without having any effects on the sintered copings.

In the same way as a tooth crown above a veneer can be manufactured.

A core (B) for an inlay can be manufactured by registering with a three dimensional optical or mechanical method the tooth prepared for an inlay (Fig. 1b) or an impression  $K_{0i}$  (Fig. 5a) from the prepared cavity. Data from the registered prepared surface guides a computer controlled milling machine for the manufacture of the body  $K_4$  (Fig. 5b) from a blank  $K_1$  (Fig. 3a) e.g. in plaster, graphite or a polymer. When the scanned impression from the inlay is enlarged taking into account both the shrinkage and the gap for cement,  $L'_4$  (Fig 5b) is obtained. From this surface  $L_4$  is calculated.  $L_4$  is the outer surface of the inlay, i.e. the surface at which porcelain can be fired in order to get the inlay ready.  $K_5$  is manufactured in e.g. plaster or some easily machined shape resistant material.

In the same way, as during the manufacturing of a core for a tooth crown an impression X for an inlay in some viscous elastic material (Fig. 4) can be manufactured. This impression (X) is then placed in the press in the same way, as described in Fig. 4 after the powder has been placed in the cavity (M) between the enlarged model ( $K_4$ ) and the impression X. During the pressing procedure the powder is compacted against the body  $K_4$ . After the pressing procedure  $K_4$  is repositioned in the computer controlled milling machine. The outer surface of the inlay ( $L'_4$ ) is machined after the repositioning in the milling machine.

The compacted body is presintered on the enlarged plaster model  $K_4$ . The shape of  $L_4$  gives the outer shape of the core of the inlay. Against this surface porcelain can be fired. In order to increase the aesthetics of the inlay the thickness of the core can be thinnest at the preparation border in order to be able to hide the core after the porcelain firing, if desired. During the modelling of the porcelain powder against the core, this is placed in the inlay preparation in the original model of the prepared tooth.

The core is generally placed in a pinned and sawed model from the whole jaw.

5 The inlay is made in a conventional way with porcelain powders. During firing the inlay is removed from the model and is fired in a porcelain furnace. Several firings with subsequent adjustments may be needed before the inlay is ready for final adjustment. Against the core dental polymers can also be attached. These polymers then will give the inlay its final shape. It is an advantage if the outer surface of the core is silanized before the polymer is joined together with the core. Finally, it is possible to give L4 such a shape that the inlay contains only densely sintered ceramic material. It is then important that the outer surface, which has contact with other teeth, is polished so that the roughness of the surface will not give wear damage on teeth in the opposite jaw.

10 A bridge can be manufactured by first registering with a three dimensional optical or mechanical method the prepared surfaces of the supporting teeth and intermediate part from a model in plaster (Fig. 6) or directly in the mouth. Prepared teeth and intermediate part of the jaw are reproduced considering the sintering shrinkage in an enlarged size with e.g. a computer controlled milling machine (Fig. 7). At the same time a gap for cement is calculated in the same way as for single crowns. Before the enlarged model is scanned, a pontics can be built up e.g. in wax on the fixture between C1 and C2. This substructure can also be calculated in the computer. Such a substructure can be seen in Fig. 7. The material in the enlarged models can be e.g. plaster, graphite or a polymer.

25 The compaction of the ceramic powder to a bridge is made in a similar way as for copings of crowns and inlays. When manufacturing a bridge an impression X is made, which leaves space for the powder both for a beam connecting the two copings and the two copings. The principal appearance

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of the body K7, which is used for manufacturing of an impression X for manufacturing of a core of a bridge in densely sintered ceramic material, is shown in Fig. 8. The impression X is made in the same way, as is described for  
5 single cores for tooth crowns. In the empty space, which surrounds C1, C2 and the intermediate gingiva, ceramic powder is placed, whereupon the tool X is placed in a pressing tool in the same way as for a single crown and a pressure Pk is applied. The ceramic powder will be compacted against  
10 C1 and C2 and against that part of the enlarged model, which is between C1 and C2. After repositioning in the milling machine the outer surface of the compacted body is machined and the core is presintered in the same way as single copings. Before presintering the support, i.e. the  
15 enlarged model described in Fig. 7, can be cut at the lines l1 and l2 in order not to break the core during presintering if plaster is used in the carrier. This division can be made before or after the compacting of the powder. It is  
20 important that the three parts are fixed during compacting of the powder in order to maintain their mutual positions. After presintering the core is adjusted, especially on the parts which could not be machined before presintering. This final adjustment can be made with a hand piece whereafter the core is sintered.

25 A bridge can also be made, which is based on supporting teeth, which have a preparation on the buccal surface for a veneer or a preparation in the lingual side for a lingual plate. The manufacturing of such a bridge will be the same as has been described above for a bridge with supporting  
30 teeth prepared for crowns.

## CLAIMS

1. Method of manufacturing an artificial tooth restoration comprising a ceramic, densely sintered high strength individual core (B) with dental porcelain (A) by powder metallurgical manufacturing methods
- 5 c h a r a c t e r i s e d in that the inner surface (I) of the core (B), which will fit against one or more prepared tooth surfaces (P) or artificial abutments are manufactured by forming a ceramic powder mixture against a surface of a
- 10 body at which said surface is manufactured by registering the surfaces of the prepared teeth or artificial abutments and their mutual relationship with a three dimensional optical or mechanical reading method either directly in the mouth or on a model in e.g. plaster after which the registered surfaces are reproduced in an enlarged size e.g. with
- 15 a computer controlled milling machine at which the enlargement is calculated from the shrinkage of the ceramic material during sintering to full density with adjustment of (I) considering desired gap for cement.
- 20 2. Method according to claim 1
- c h a r a c t e r i s e d in that the core is manufactured by dry pressing a ceramic powder with a rubber tool X made from some viscous elastic material, close to desired size against the enlarged preparations K<sub>2</sub>, K<sub>4</sub> and K<sub>6</sub> made by
- 25 milling of premade plaster, graphite or polymer blanks.
3. Method according to claim 2
- c h a r a c t e r i s e d in that the rubber tool X is made by taking an impression of the body K<sub>3</sub>, K<sub>5</sub> or K<sub>7</sub>.
4. Method according to claims 2 and 3
- 30 c h a r a c t e r i s e d in that after compacting of the ceramic powder against K<sub>2</sub>, K<sub>4</sub> or K<sub>6</sub>, K<sub>2</sub>, K<sub>4</sub> or K<sub>6</sub> are repositioned in the milling machine at which the outer contour of the core is milled to desired shape, whereupon the core is presintered on K<sub>2</sub>, K<sub>4</sub> or K<sub>6</sub>.

5. Method according to claims 2, 3 and 4  
characterised in that the presintered core is removed from K<sub>2</sub>, K<sub>4</sub> or K<sub>6</sub> and is sintered to full density.

6. Method according to claims 1, 2, 3 and 4  
5 characterised in that the core material consists of high strength, densely sintered ceramic material with a relative density of >99%.

7. Method according to claim 5  
characterised in that the ceramic material in  
10 the core is based on one or more of the oxides Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, MgO, ZrO<sub>2</sub> or ZrO<sub>2</sub> with up to 25 mole% Y<sub>2</sub>O<sub>3</sub> or MgO.

8. Method according to claims 5 and 6  
characterised in that the core material also comprises whiskers and/or particles of SiC, TiN, ZrO<sub>2</sub>  
15 and/or ZrN.



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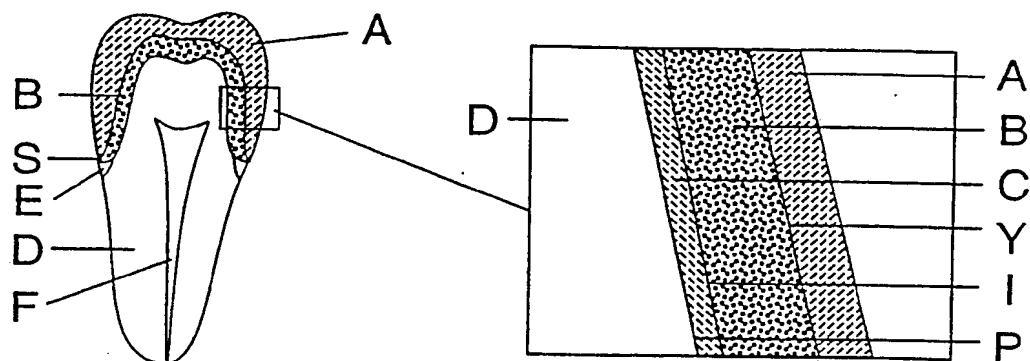


Fig. 1a

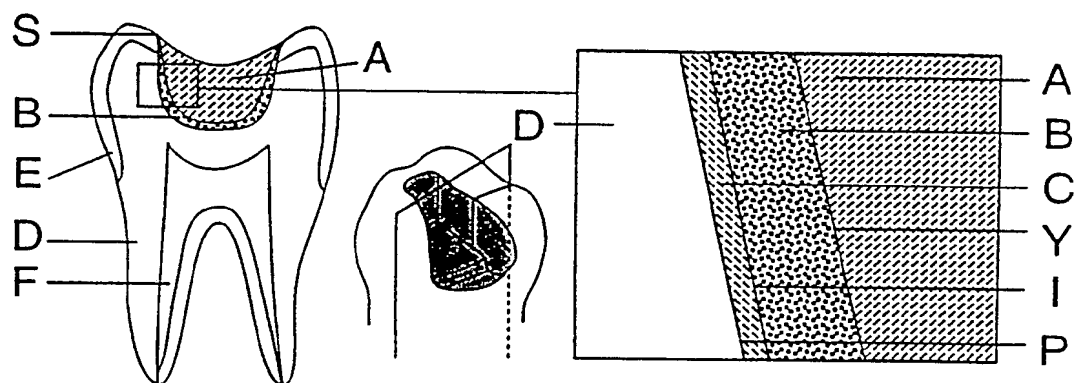


Fig. 1b

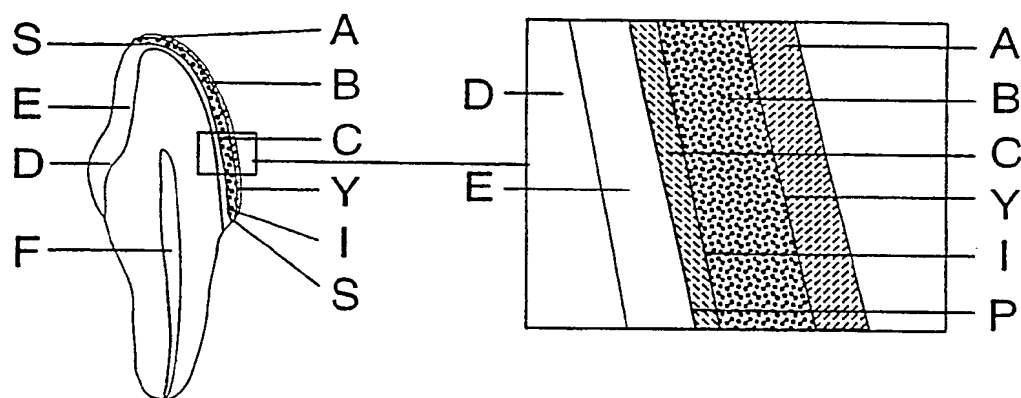
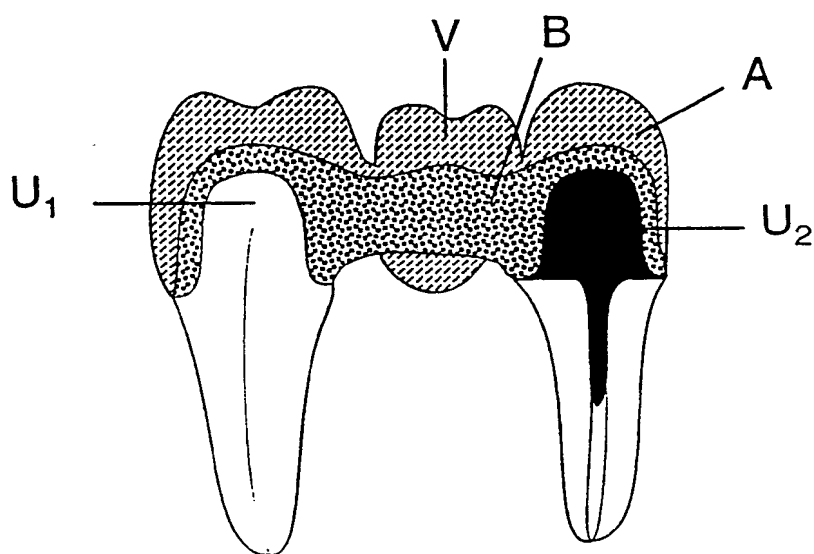


Fig. 1c

Fig. 1

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**Fig. 2**

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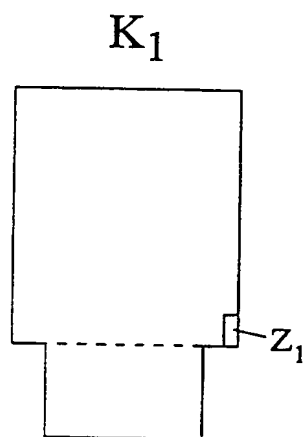


Fig. 3a

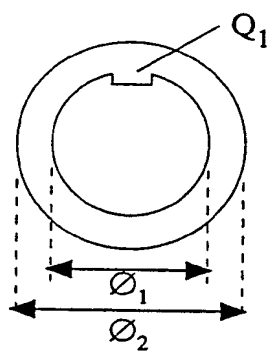


Fig. 3b

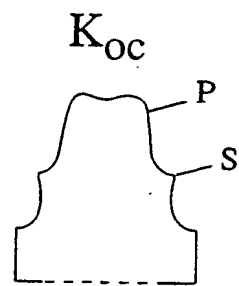


Fig. 3c

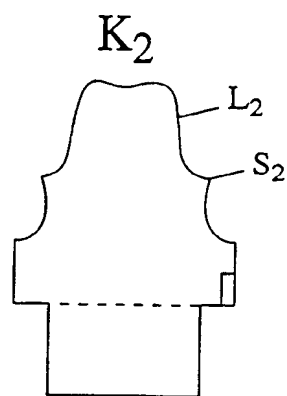


Fig. 3d

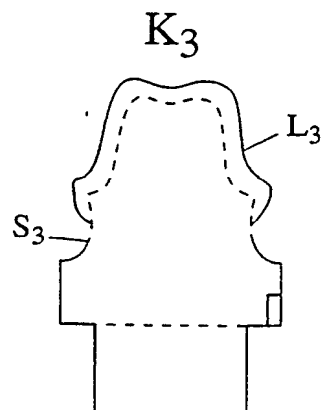


Fig. 3e

**Fig. 3**

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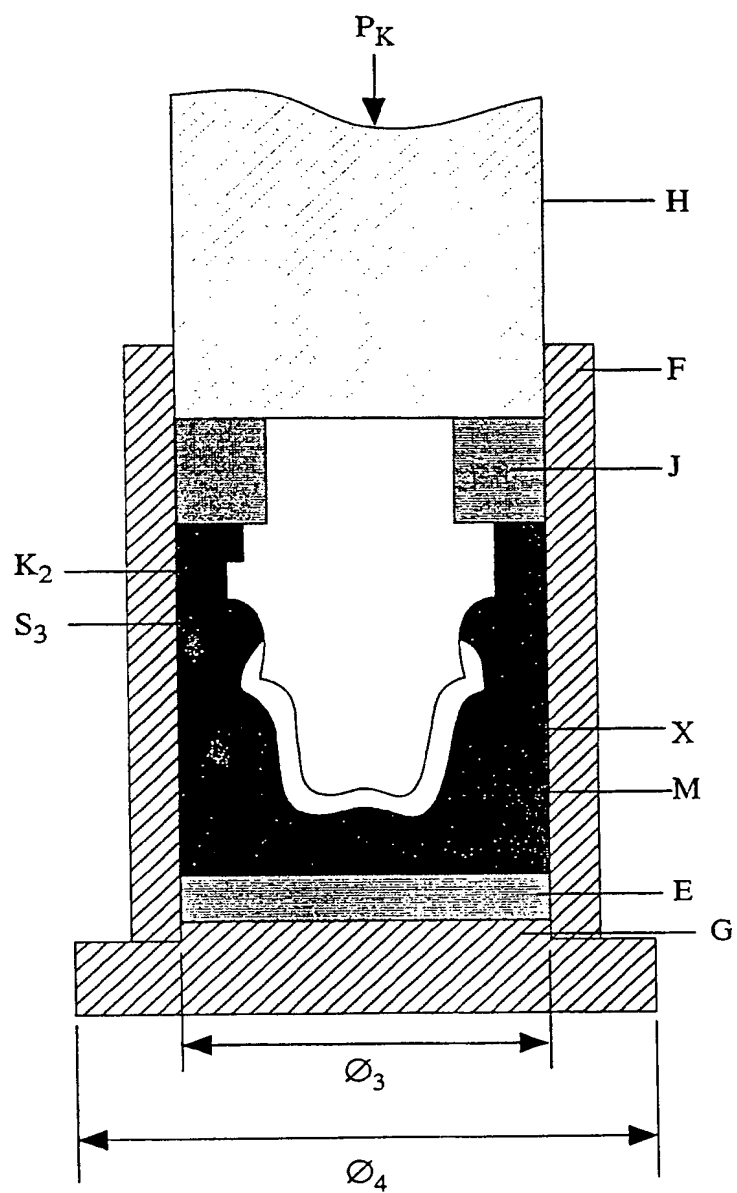


Fig. 4

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$K_{oi}$

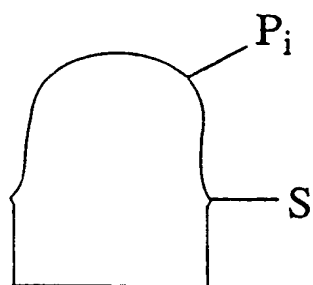


Fig. 5a

$K_4$

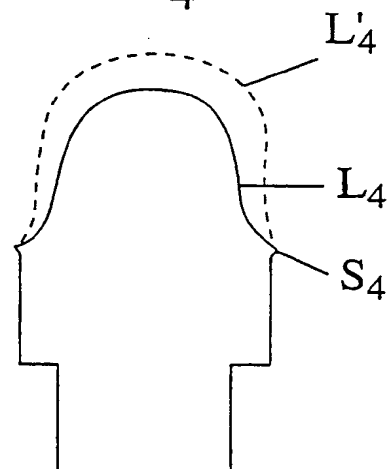


Fig. 5b

$K_5$

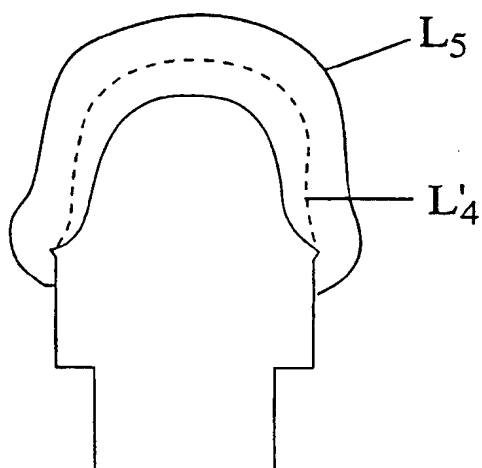


Fig. 5c

**Fig. 5**

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Kob

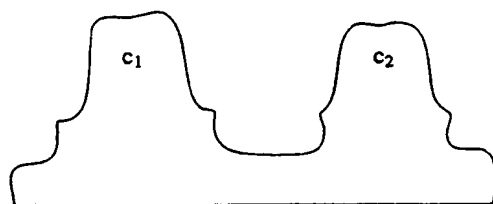


Fig 6

K6

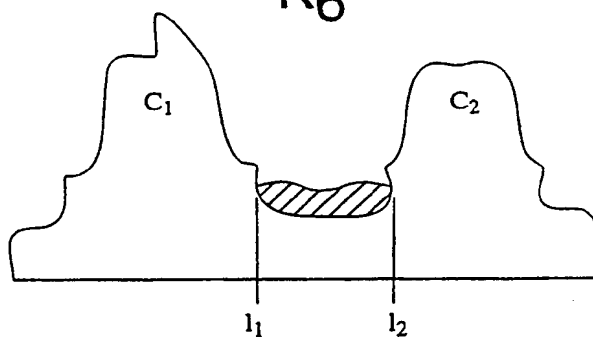


Fig 7

K7

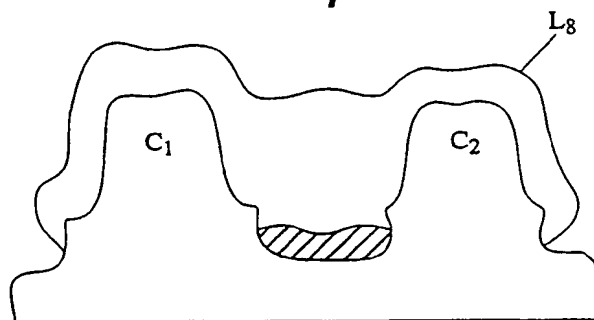


Fig 8

1  
INTERNATIONAL SEARCH REPORT

International application No.  
PCT/SE 94/00507

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC5: A61C 5/00, A61C 5/10, A61C 13/00  
According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC5: A61C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 5080589 (AGNETA E. ODEN ET AL), 14 January 1992 (14.01.92)  --	1-8
A	US, A, 5106303 (AGNETA E. ODEN ET AL), 21 April 1992 (21.04.92)  -- -----	1-8

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

- |  |   |
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| <p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> | <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&amp;" document member of the same patent family</p> |
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Date of the actual completion of the international search

2 Sept 1994

Date of mailing of the international search report

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**INTERNATIONAL SEARCH REPORT**  
Information on patent family members

02/07/94

International application No.  
PCT/SE 94/00507

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US-A-	5080589	14/01/92	EP-A-	0375647	27/06/90
US-A-	5106303	21/04/92	DE-D,T-	69004306	03/03/94
			EP-A,B-	0389461	26/09/90

Form PCT/ISA/210 (patent family annex) (July 1992)